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Correlation of mechanical properties in bulk metallic glasses with ^{27}Al NMR characteristics

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We report ^{27}Al NMR and magnetic susceptibility measurements of Zr and ZrHf-based bulk metallic glasses (BMGs). ^{27}Al NMR Knight shift shows that there exists a clear correlation between the local electronic properties at Al sites and mechanical properties. In addition, magnetic susceptibility measurements also provide clues on the influence of the electronic states, especially the strong influence of *d*-orbital characteristics on the mechanical properties of toughness and hardness.

bulk metallic glass, nuclear magnetic resonance, knight shift, magnetic susceptibility, mechanical properties

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Many families of multi-component bulk metallic glasses (BMGs) have been studied extensively due to their good glass forming ability (GFA) [1–6], thermal stability, as exhibited by their wide supercooled region [2,5,7], and favorable mechanical properties such as high yield strength [5,6]. Earlier studies have discussed correlations of GFA with thermodynamics [8,9], kinetics [8–10], electronic structure [11], and atomic structures [12–14]. To optimize the potential of metallic glasses for engineering applications, an understanding of correlations between electronic structures and mechanical properties can be quite valuable for designing and fine-tuning the properties of BMGs. Previous experimental [15–17] and computational studies of transition metal (TM)-based BMGs [16,18,19] have addressed the issue of the relationship between electronic structures and mechanical properties such as strength and ductility. More experimental evidence would be very useful for further establishing and understanding the correlations between structural properties and electronic properties of BMGs. In this study, we use ^{27}Al NMR and magnetic susceptibility to

show strong correlations between hardness and toughness of Zr and ZrHf-based BMGs and local electronic structures.

1 Experimental

In this study, Zr-based BMGs ($\text{Zr}_{56}\text{Co}_{28}\text{Al}_{16}$, $\text{Zr}_{60}\text{Ni}_{21}\text{Al}_{19}$, $\text{Zr}_{48}\text{Cu}_{45}\text{Al}_7$, $\text{Zr}_{60}\text{Cu}_{28}\text{Al}_{12}$, and $\text{Zr}_{61}\text{Ti}_2\text{Cu}_{25}\text{Al}_{12}$) [3] and ZrHf-based BMGs ($(\text{Zr}_{1-x}\text{Hf}_x)_{52.5}\text{Ti}_5\text{Cu}_{17.9}\text{Ni}_{14.6}\text{Al}_{10}$ from $x = 0.0$ to 1.0) [4] were used for investigation due to their good GFA and favorable mechanical properties such as toughness and hardness. Details regarding the BMG sample preparations and mechanical testing methods and properties were reported in [3] and [4]. ^{27}Al NMR experiments were performed at room temperature in a magnetic field of 8.89 T. 1.0 mol/L $\text{Al}(\text{NO}_3)_3$ aqueous solution was used as shift reference for ^{27}Al Knight shift measurements. NMR spectra were acquired using a Hahn-echo pulse sequence with a strong rf field strength of $\omega_{\text{rf}}/2\pi = 100$ kHz and recycle delay of 300 ms. Magnetization measurements were conducted at room temperature using a Quantum Design SQUID magnetometer. The magnetization of metallic glass samples were

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measured as a function of varying field from 50000 to -50000 Oe in steps of 5000 Oe. The total magnetic susceptibility for each sample was then calculated and also corrected for the weak diamagnetic background susceptibility due to an empty sample holder.

2 Results and discussion

^{27}Al NMR was used to probe local atomic and electronic structure as a function of composition. Figure 1(a) and (b) shows ^{27}Al NMR spectra for Zr and ZrHf-based BMG compositions, respectively. Changes of the local electronic states are established by the systematic changes of spectral shift caused by variations of both the aluminum and transition metal (Zr, Cu, Ni, Co, Ti) content (Figure 1(a)) and also by systematic variation of the Hf/Zr ratio with fixed aluminum content (Figure 1(b)). The dominant shift mechanism for metallic systems is given by the Knight shift K_{iso} . For transition metal alloy systems the Knight shift is expressed as $K_{\text{iso}} = K_s + K_d + K_{\text{orb}} = \alpha_s \chi_{\text{Pauli}}^s + \alpha_{\text{orb}} \chi_{\text{orb}}$ [20,21]. The most prominent contribution comes from K_s , which is the direct contact shift due to the Fermi contact hyperfine interaction associated with the Pauli susceptibility χ_{Pauli}^s of s electrons at the Fermi level. K_d is caused by transition metals due to the polarization of d -electrons by the external field and provides an indirect s - d exchange interaction with the s -conduction electrons described by $K_d = \alpha_D \chi_D$, where α_D is the hyperfine coupling constant and χ_D is the d -electron Pauli susceptibility. K_{orb} is the orbital shift contribution derived from the orbital moment induced in occupied conduction electron states, where χ_{orb} is the orbital susceptibility and α_{orb} is the coupling constant. This second order perturbation effect gives rise to magnetic shielding of the nucleus and is most important in transition metals with half-filled d -band [20]. The changes in Figure 1(a) and (b) of K_{iso} observed in both Zr and ZrHf-based systems indicate that the atomic structure at Al sites is changing with composition. The values of the Knight shift given in Figure 1 (a) and (b) are on the order of ~350 ppm demonstrating that the s -electron contribution at the Fermi energy is quite small as compared to the shift in pure Al metal (~1630 ppm) [20]. This is consistent with previous NMR studies of Al-based BMGs [15,16,22] and recent electronic structure calculations of Al-Zr-Cu [16] and Al-Ca [23] amorphous systems. The calculations indicate that the s -electron band is significantly shifted to higher binding energies whereas the valence d -electron band of transition metals remain prominent at the Fermi level [16,24].

Correlations of hardness and toughness with the ^{27}Al Knight shift K_{iso} values are displayed in Figure 2. These results demonstrate that the local electronic structure at Al sites correlate with the hardness and toughness properties of BMGs. Figure 2(b) shows linear correlation between the

hardness and the ^{27}Al Knight shift, where the Zr/Hf ratio is an implicit parameter. Figure 2 (a) shows that the toughness decreases with increasing ^{27}Al Knight shift K_{iso} although the correlation is not linear. Magnetic susceptibility measurements were obtained to further understand the change of electronic structure with toughness and hardness properties. The insets of Figure 2 (a) and (b) display a direct correlation of total magnetic susceptibility χ_{exp} with K_{iso} . The total measured magnetic susceptibility for each composition was corrected by a weighted average of the diamagnetic core susceptibilities [25]. The total susceptibility can be further expressed as $\chi_{\text{exp}} - \chi_{\text{core}} = \chi_{\text{Pauli}}^s + \chi_L + \chi_{\text{Pauli}}^d + \chi_{\text{orb}}$ [21,22,26], where χ_L is the Landau diamagnetic s -conduction electron susceptibility. The free s -conduction electron components χ_{Pauli}^s and χ_L of the total susceptibility can be calculated from $\chi_{\text{Pauli}}^s = (1.33 \times 10^{-6}) V_a^{2/3} V_e^{1/3}$ and $\chi_L = -1/3 \chi_{\text{Pauli}}^s$ [26], where V_a is the atomic volume in \AA^3 and n_e is the number of conduction elements per atom. Except for χ_{orb} , all contributions to the total susceptibility are directly related to the s or d electronic density of states (DOS). The d -electron contribution $\chi_{\text{Pauli}}^d + \chi_{\text{orb}}$ of the total susceptibility can be approximated by subtracting the free s -conduction contribution $\chi_{\text{Pauli}}^s + \chi_L$ from the total measured susceptibility.

Table 1 summarizes the results of total, d -electron, and s -electron magnetic susceptibilities in addition to the shift and mechanical properties for each Zr and ZrHf-based composition. A direct relationship between the total susceptibility (or DOS) and mechanical properties was also observed in TE-TL (TE=Zr, Hf, Ti; TL=Cu, Ni, Co) metallic glasses [17,27] where the hardness and Young's modulus E were found to increase in proportion to the TL content while resulting in a decrease of the total DOS. Table 1 also reveals that the d -electron contribution to the magnetic susceptibility for both alloy systems is significant comprising approximately half of the total magnetic susceptibility. This is compatible with X-ray and ultraviolet photoemission studies [24,28] of TE-TL (TE=Zr, Ti; TL=Cu, Ni, Co, Fe) metallic glass alloys that show the DOS at the Fermi level is dominated by d -electron valence band states of TE elements. For both Zr and ZrHf-based BMG systems, the d -electron susceptibility $\chi_{\text{Pauli}}^d + \chi_{\text{orb}}$ (or DOS) is proportional to the toughness and hardness. One should note that $\text{Zr}_{56}\text{Co}_{28}\text{Al}_{16}$ does not fit into this trend and can be attributed to the susceptibility contribution caused by χ_{orb} [20] due to a nearly half-filled d -band of Co, which is otherwise negligible in the remaining compositions of this study. The dependence of toughness and hardness properties on d -electron susceptibility is demonstrated in Figure 3, where the outlier from $\text{Zr}_{56}\text{Co}_{28}\text{Al}_{16}$ is clearly noted. Furthermore, while the ^{27}Al Knight shifts demonstrate that local electronic structure at Al-sites is strongly coupled to mechanical properties of metallic glass, Figure 3 shows that the d -electron susceptibility provides a strong correlation with hardness and toughness properties.

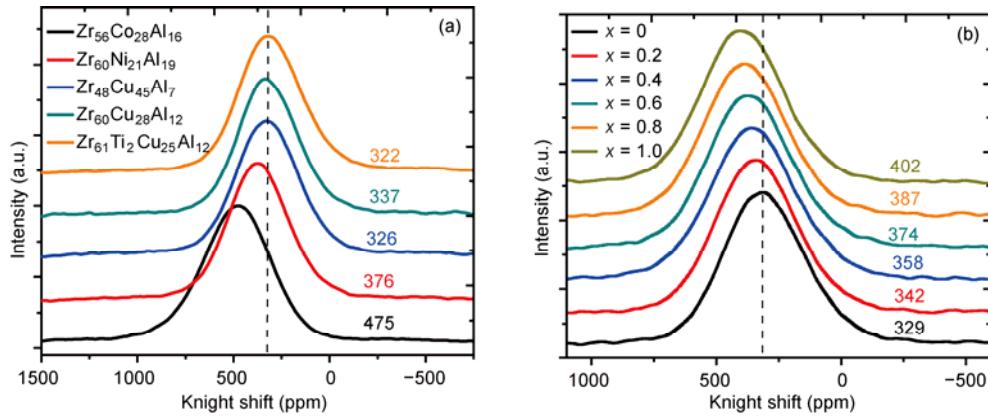


Figure 1 (a) Room temperature ^{27}Al NMR spectra for $\text{Zr}_{56}\text{Co}_{28}\text{Al}_{16}$, $\text{Zr}_{60}\text{Ni}_{21}\text{Al}_{19}$, $\text{Zr}_{48}\text{Cu}_{45}\text{Al}_7$, $\text{Zr}_{60}\text{Cu}_{28}\text{Al}_{12}$, and $\text{Zr}_{61}\text{Ti}_{2}\text{Cu}_{25}\text{Al}_{12}$ BMGs. A dashed line centered at 322 ppm serves as a guide for the eyes. (b) Room temperature Al NMR spectra for $(\text{Zr}_{1-x}\text{Hf}_x)_{52.5}\text{Ti}_5\text{Cu}_{17.9}\text{Ni}_{14.6}\text{Al}_{10}$ BMG system from $x = 0$ to 1.0. A dashed line centered at 329 ppm for $x = 0$ serves to demonstrate systematic changes of the shift with composition.

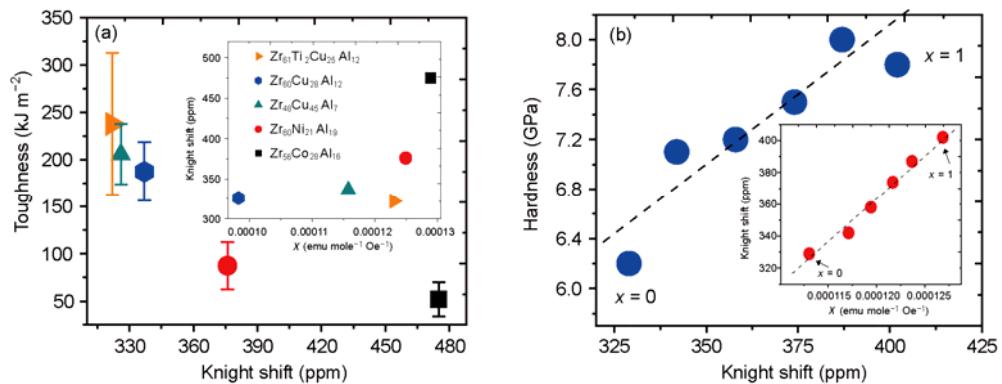


Figure 2 (a) Toughness (kJ m^{-2}) properties are plotted as a function of Knight shift K_{iso} for Zr-based BMGs [3] demonstrating a direct correlation between mechanical properties and local electronic structure. (b) Hardness (GPa) properties for ZrHf-based BMGs [4] are plotted versus the Knight shift K_{iso} showing a systematic correlation. The insets of both figures display the corresponding correlation between the Knight shift K_{iso} and the total magnetic susceptibility $\chi_{\text{exp}} - \chi_{\text{core}}$.

Table 1 Room temperature ^{27}Al Knight shifts, total magnetic susceptibility, s -electron susceptibility, d -electron susceptibility, and toughness and hardness mechanical properties for Zr [3] and ZrHf-based [4] BMGs

Sample	Knight shift (ppm)	$\chi_{\text{exp}} - \chi_{\text{core}}$ $\left(\frac{\text{emu}}{\text{mole Oe}} \right) \times 10^{-4}$	$\chi_s + \chi_L$ $\left(\frac{\text{emu}}{\text{mole Oe}} \right) \times 10^{-5}$	$\chi_d + \chi_{\text{orb}}$ $\left(\frac{\text{emu}}{\text{mole Oe}} \right) \times 10^{-5}$	Toughness (kJ m^{-2})
$\text{Zr}_{56}\text{Co}_{28}\text{Al}_{16}$	475	1.30	7.02	5.87	52
$\text{Zr}_{60}\text{Ni}_{21}\text{Al}_{19}$	376	1.25	7.82	4.67	87.5
$\text{Zr}_{48}\text{Cu}_{45}\text{Al}_7$	326	0.98	4.50	5.32	206
$\text{Zr}_{60}\text{Cu}_{28}\text{Al}_{12}$	337	1.16	6.32	5.26	187.5
$\text{Zr}_{61}\text{Ti}_{2}\text{Cu}_{25}\text{Al}_{12}$	322	1.23	6.64	5.67	238
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$(\text{Zr}_{1-x}\text{Hf}_x)_{52.5}\text{Ti}_5\text{Cu}_{17.9}\text{Ni}_{14.6}\text{Al}_{10}$					
x					Hardness (GPa)
0	329	1.13	5.97	5.33	6.2
0.2	342	1.17	6.24	5.47	7.1
0.4	358	1.19	6.46	5.48	7.2
0.6	374	1.22	6.66	5.51	7.5
0.8	387	1.24	6.82	5.54	8
1	402	1.27	6.97	5.71	7.8

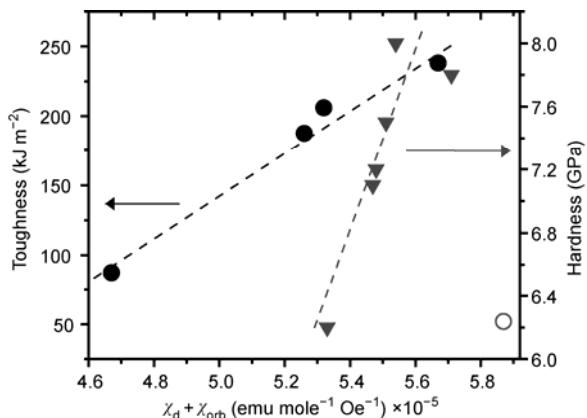


Figure 3 Toughness (●) and hardness (▼) properties for Zr and ZrHf-based BMGs are plotted versus $\chi_d + \chi_{\text{orb}}$ from Table 1 showing an approximately linear correlation. The outlier of the trend seen here is from $\text{Zr}_{56}\text{Co}_{28}\text{Al}_{16}$ due to enhanced χ_{orb} and is noted with a different symbol (○).

Previous electronic structure studies of Zr-Cu-based metallic glasses [3,16] attribute a decreasing toughness (or an increase in brittleness) to an enhancement of covalent-like bonding, or specifically an increase in Al-TM *sp-d* hybridization in band structure. While this cannot be confirmed directly here, the implications of the data presented in Table 1 are that both the *s* and *d* magnetic susceptibilities of BMGs are highly influential for obtaining favorable mechanical properties. This can be rationalized in terms of the cohesive energies or interatomic bond strengths in transition metal-based alloy systems [29] and was briefly alluded to in recent studies of Zr and ZrHf-based BMGs [4,17]. The cohesive properties of alloys depend directly on the densities of *s* and *d*-electron states at the Fermi energy and the *d*-electron valence band width [29]. This is also important in achieving large exothermic heats of alloy formation [28,30] which are significant for good GFA and thermal stability [4, 17]. Large *d*-electron valence differences between constituents in an alloy (i.e. Zr-Cu) have been experimentally observed to induce significant *d*-band splitting that enhances the *d*-electron valence band width and density of states [24], which thereby increases the cohesive energy [29]. This is consistent with the data presented in Table 1 where the largest toughness value is observed in $\text{Zr}_{61}\text{Ti}_2\text{Cu}_{25}\text{Al}_{12}$ whereas it is the smallest in $\text{Zr}_{56}\text{Co}_{28}\text{Al}_{16}$ due to a much smaller valence difference between Zr and Co resulting in minimal *d*-band splitting [24]. Similarly, in ZrHf-based alloys where the cohesive energy due to the *s* and *d*-band of Hf is significantly higher than Zr [29], the hardness increases systematically from $x = 0$ to $x = 1.0$ due to a larger Hf/Zr fractional content.

3 Conclusions

In conclusion, this work provides insight into the correlations that exist between both the local electronic structure at

Al sites and magnetic susceptibility with hardness and toughness properties of Zr and ZrHf-based BMGs. The NMR and magnetic susceptibility results discussed also show consistency with previous studies of BMGs. The experimental data presented here clearly provides evidence that for transition metal-based BMGs, the *d*-electron structure is very important for obtaining favorable mechanical properties. The correlations observed in this study provide a guide for designing BMGs with specific mechanical properties of interest.

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